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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: Sameer D. Mehta, et al.	)
SERIAL NUMBER: 10/027,742	) **EXAMINER: R.A. Lee**
FILING DATE: December 20, 2001	) **ART UNIT: 1713**
FOR: Ethylene Polymer Compositions Having Improved Melt Strength	)

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DECLARATION UNDER 37 CFR 131

We, Sameer D. Mehta, Harilaos Mavridis, Venki Chandrashekar and Edward S. Vargas, declare and say as follows:

1. We are the Applicants/coinventors named in the above-identified application which discloses and claims ethylene polymer composites having improved melt strength comprising: (a) 76 to 99.25 weight percent, based on the weight of the total composition, of an ethylene homopolymer or ethylene-C_{3-8} α-olefin copolymer base resin; (b) 0.5 to 12 weight percent of an organically modified clay consisting of a smectite clay that has been ion-exchanged and intercalated with a dimethyl dihydrogenated tallow quaternary ammonium ion and (c) 0.25 to 12 weight percent of an ethylene polymer compatibilizing agent selected from the group consisting of ethylene-vinyl carboxylate copolymers and polymers of ethylene having from 0.1 to 8 weight percent ethylenically unsaturated carboxylic acid or derivative monomer copolymerized or grafted; the weight ratio of (b) to (c) ranging from 1:5 to 1:0.1.

2. We are aware of the publication of Ki Hyun Wang, et al., Polymer 42 (2001), 9819-9826, entitled "Synthesis and Characterization of Maleated Polyethylene/Clay Nanocomposites," a copy of. which is annexed hereto as Exhibit 1. This article was published in the November 2001 issue of Polymer and the article was available on-line August 22, 2001 as evidenced by Exhibit 2. To the best of our knowledge, August 22, 2001 is the earliest date of availability of the Wang, et al., article.

3. As evidence of our conception and reduction to practice prior to the August 22, 2001 date, attached and incorporated into this Declaration are Exhibits 3-8 which are true and accurate copies of original paper and electronic records, with dates blacked out, generated during the course of our work to develop the ethylene polymer composites having improved melt strength and describing experiments performed by us or under our supervision. All of said records bear dates prior to the August 22, 2001 date of the Wang, et al., article.

4. Exhibit 3 is a copy of an electronic memo sent to our technician, Dan Riopell, identifying six (6) masterbatch formulations, six (6) high density polyethylene (HDPE) let down formulations and two (2) controls for preparation. The let down formulations (identified as HDPE-1 thru HDPE-6) all contained 6 weight percent dimethyl dihydrogenated tallow quaternary ammonium ion and 3 weight percent compatibilizing agent (CMB 24, AX 8900 or UE 634). A small amount of silane coupling agent (A-1100) was also included in the formulations. The control resins contained no masterbatch and therefore contained no organically modified clay or compatibilizing agent.

5. Exhibit 4 is a summary of the run conditions used by Dan Riopell to melt blend and extrude HDPE-1 thru HDPE-6 and the two control resins. A ZSK-30 twin screw extruder was used and Z1-Z9 indicate the temperature (ºC) of the nine (9) heating zones.

6. Exhibit 5 is a printout of the data generated by the physical and analytical testing laboratory for blends HDPE-1 thru HDPE-6 and the two control resins. The data were entered into the company sample manager computer base in accordance with the standard practice. The samples are identified under the heading "Sample Name." As part of the analytical testing, the samples were rheologically evaluated utilizing a dynamic oscillatory rate sweep (DORS). From the rheological data, it was concluded that the extrusion conditions, primarily the screw configuration, were too severe resulting in degradation. As a result, the blends and controls were run using less severe process

conditions, namely, a different screw configuration and slightly lower temperature profile, designed to reduce the amount of shear and heat in the extruder. These corresponding new blends were identified as HDPE-7 thru HDPE-12 and the controls identified as HDPE-C1-ZSK 30 and HDPE-C2-ZSK 30. Several other resins were included in this run. The blend compositions and control resins are identified in Exhibit 6 and a notation at the bottom of the table indicates the blends are the "same as HDPE 1-6 except that the let down are done on a less harsh screw on ZSK-30." Run conditions are set forth in Exhibit 7. A handwritten entry, made on the run sheets at the time the runs were made identified the various samples as HDPE-7 thru HDPE-12. The blends HDPE-7 thru HDPE-12 were also identified on the run sheets as "HDPE-1 with soft screw" thru HDPE-6 with soft screw."

7. Exhibit 8 is a printout of the data generated by the physical and analytical testing laboratory for blends HDPE-7 thru HDPE-12 and the two control resins. The data were entered into the company sample manager computer base in accordance with the standard practice. The samples are identified under the heading "Sample Name." As part of the analytical testing, the samples were rheologically evaluated utilizing a dynamic oscillatory rate sweep (DORS). Based on the rheological data, the complex viscosity of the samples was calculated and a noticeable increase in complex viscosity with decreasing frequencies was observed versus the controls. This increase indicates the composite blends HDPE-7 thru HDPE-12 have superior melt strength compared to the HDPE control resins.

8. As a result of the improved melt strength observed for HDPE-7 thru HDPE-12, an Invention Disclosure was prepared and submitted to our Legal Department. A copy of the invention disclosure is annexed hereto as Exhibit 9.

9. As a further result of the success with the HDPE formulations, work on the project continued and this approach was utilized to determine if melt strength improvement of other resins could be achieved. One aspect of this on-going work involved preparation and evaluation of linear low density polyethylene (LLDPE) formulations. LLDPE formulations were prepared and evaluated in the same manner as the HDPE blends. Test results obtained for LLDPE containing 6 weight percent organically modified clay and 3 weight percent compatibilizer along with results for the LLDPE control resin are annexed hereto as Exhibit 10.

10. As a result of the improved melt strength obtained with the LLDPE formulated with the organically modified clay and compatibilizing agent, a second Invention Disclosure was prepared and submitted to our Legal Department. A copy of that Invention Disclosure is annexed hereto as Exhibit 11.

11. Our work to develop ethylene polymer composites having improved melt strength utilizing organically modified clays and compatabilizing agents continued with diligence from prior to August 22, 2001 until the time of filing our above-identified patent application.

We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed: SMehta 06/25/03
Sameer D. Mehta Date

Signed: HMavridis 6/25/03
Harilaos Mavridis Date

Signed: Venki Chandrashekar 6/26/03.
Venki Chandrashekar Date

Signed: Edward S. Vargas 6/25/03
Edward S. Vargas Date

Exhibit 1

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Polymer 42 (2001) 9819–9826

polymer

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Synthesis and characterization of maleated polyethylene/clay nanocomposites

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Abstract

Maleic anhydride grafted polyethylene (maleated polyethylene)/clay nanocomposites were prepared by simple melt compounding. The exfoliation and intercalation behaviors depended on the hydrophilicity of polyethylene grafted with maleic anhydride and the chain length of organic modifier in the clay. When the number of methylene groups in alkylamine (organic modifier) was larger than 16, the exfoliated nanocomposite was obtained, and the maleic anhydride grafting level was higher than about 0.1 wt% for the exfoliated nanocomposite with the clay modified with dimethyl dihydrogenated tallow ammonium ion or octadecylammonium ion. The pure LLDPE showed only the intercalation, which does not depend on the initial spacing between clay layers.

Keywords: Polyethylene; Nanocomposite; Melt compounding

1. Introduction

Polymer nanocomposites are a class of hybrid materials composed of organic polymer matrix in which inorganic particles with nanoscale dimension are imbedded [1–6]. At this scale, the inorganic fillers improve dramatically the physical and mechanical macroscopic properties of polymer even though their amount is small. The polymer nanocomposite exhibits higher heat distortion temperatures, enhanced flame resistance, increased modulus, better barrier properties and decreased thermal expansion coefficient [7–8]. The enhanced properties are presumably due to the synergistic effects of nanoscale structure and interaction of fillers with polymer. Because of many advantages of the nanocomposites, polymer/clay nanocomposites have been intensely investigated [9].

Polyethylene is one of the most widely used polyolefin polymers. Since it does not include any polar group in its backbone, it is thought that the homogeneous dispersion of the clay minerals in polyethylene is not realized. In general, the clay is modified with alkylammonium to facilitate its interaction with a polymer because the alkylammonium make the hydrophilic clay surface organophilic. However, the organically modified clay does not disperse well in the nonpolar polypropylene or polyethylene since such non-polar polymers are still too hydrophobic [10–12]. Jeon and coworkers reported the intercalated morphology of HDPE nanocomposites prepared by blending HDPE with sodium montmorillonite cation-exchanged with protonated dodecylamine in solution [10]. However, the presence of fairly large stacks indicated poor dispersion. Only when in situ polymerization was performed, polyethylene/clay nanocomposite showed exfoliated morphology [12–14].

Initial attempts to create the nonpolar polymer/clay nanocomposites by simple melt mixing were based on the introduction of a modified oligomer to mediate the polarity between the clay surface and polymer [15–23]. One of the typical examples is polypropylene/clay nanocomposite system given by Toyota. They used polypropylene oligomer modified with about 10 wt% of maleic anhydride (MA) as compatibilizer and clays exchanged with stearylammonium cation to obtain exfoliated or semiexfoliated clay morphology [17,18,21–23]. In the nanocomposite system with three components, it was pointed out that the miscibility between maleated oligomer and matrix polymer played a key role in composite properties.

Various aspects of polymer/organoclay hybrids obtained by melt intercalation have been investigated in recent years: interlayer structure of modified clays [24], structural evolution during the intercalation [25], mobility of intercalated polymer chains described by both the glass transition behavior [26,27]. The effects of factors such as the length and number of alkyl groups of cationic modifier molecule and the polarity of matrix polymer on the intercalation behavior were also investigated [28]. Some attempts to

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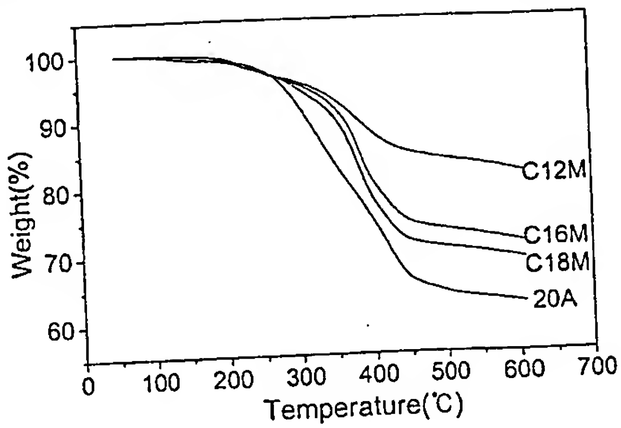

Fig. 1. TGA curve of organophilic clay.

predict the phase diagram and structural characteristics of nanocomposites were performed using thermodynamics based on the lattice model [29], the Onsager based approach [30], the self-consistent field (SCF) theory [31,32] and the molecular simulation method [33–35]. It has been reported that the intercalation of organoclay by polystyrene (PS) takes place in very short time and the equilibrium gallery height is independent of molecular weight of PS, even though the intercalation rate is slower when the molecular weight is higher [27,28].

The equilibrium structure of polymer/layered silicate nanocomposites, in particular with organically modified layered silicates, have been shown to be a strong function of the nature of a polymer (polar or apolar), the charge density of a layered silicate, as well as the chain length and structure of a cationic surfactant.

Balazs et al. have considered the SFC in order to investigate the factors promoting the penetration of polymers into layered silicates [31,36]. They first varied properties related to the nature of tethered surfactant chains. They found out that an increase in the surfactant length (approaching the length of the polymer chains) improved the layers separation by allowing the polymer to adopt more conformational degrees of freedom. On the contrary, increase in the length of polymer chains tends to render the interlayer mixture immiscible. They also reported the effect of the surfactant density on the intercalation process, showing that excessive density of tethered alkyl chains could impede the formation of intercalated structures.

In this study, we investigate the effect of alkylammonium modifier to clay (montmorillonite) and the maleic anhydride (MA) grafted level of polyethylene on morphology of maleated polyethylene/clay nanocomposite prepared by simple melt compounding. The alkylammonium chain length may change the degree of interaction between clay and polyethylene. We also report the intercalation behavior of pure polyethylene, which is not modified with MA, into organically modified montmorillonite.

2. Experimental

2.1. Materials

'Kunipia F' supplied by Kunimine Ind. Co. was a Na + type montmorillonite, with a cation exchange capacity of 119 meq/100 g. Dodecyl, hexadecyl and octadecylamine purchased from Aldrich were used as organic modifier of MMT. Maleic anhydride (MA) and dicumylperoxide (DCP) were used as the modifier of polyethylene and a radical initiator, respectively.

The modified montmorillonite (Closite 20A, abbreviation: 20A) supplied by Southern Clay Products was used, which was ion-exchanged with dimethyl dihydrogenated tallow ammonium ions. (Tallow was composed predominantly of octadecyl chains with smaller amount of lower homologues. The approximate composition was C_{18} 65%, C_{16} 30% and C_{14} 5%.)

Maleic anhydride modified polyethylene (PEMA, 0.85 wt% maleic anhydride grafted, Aldrich) and linear low density polyethylene (LLDPE) from HanWha Chemical Co. were used. All chemicals were used without further purification.

2.2. Preparation of organically modified MMT

Organophilic MMT was prepared using various alkyl ammonium salts according to the reported methods [4,22]. For example, the modification of montmorillonite by octadecylamine was carried out as follows. Sodium montmorillonite (80 g; 119 meq/100 g cation exchange capacity) was dispersed into 5000 ml of hot water (80°C) with continuous stirring. Octadecylamine (31.1 g, 115 mmol) and conc. hydrochloric acid (11.5 ml) were dissolved into 2000 ml of hot water (80°C). It was poured into the hot montmorillonite–water solution under vigorous stirring for 5 min to yield a white precipitate. The precipitate was collected on a cloth filter, washed three times with 2500 ml of hot water (80°C), and freeze-dried to yield a modified MMT with octadecylammonium. This organically modified MMT was termed C18M. The modifications of MMT with hexadecylamine and dodecylamine were similar to the case of octadecylamine except that 71.7 g of hexadecylamine and 55 g of dodecylamine were used instead of 80 g of octadecylamine. The modified MMTs with hexadecylamine and dodecylamine were named as C16M and C12M, respectively.

2.3. Preparation of PEMA/clay nanocomposites and maleic anhydride grafted polyethylene by reactive extrusion

Several types of nanocomposites with different compositions of the organically modified clays and maleated polyethylene were prepared by melt compounding at 140°C, using Brabender mixer with the chamber size of 50 cm^3. Screw speed was 60 rpm and the mixing time was 20 min for all the cases.

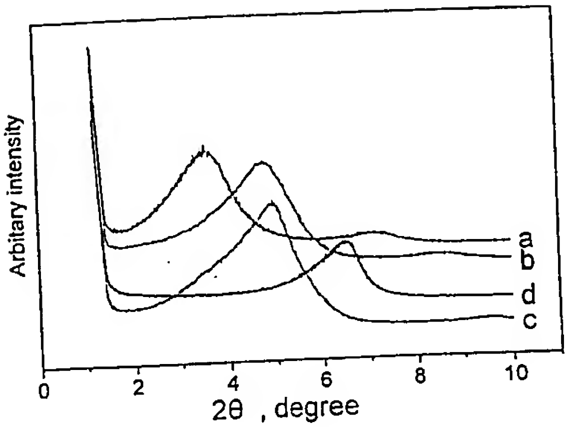

Fig. 2. XRD patterns of organophilic clays: (a) 20A, (b) C18M, (c) C16M, (d) C12M.

We prepared two types of maleated polyethylenes (maleated PE) in twin screw extruder (Gottfert Corp., screw diameter: 19 mm) at a constant rotating speed of 30 rpm and the barrel ($L/D=25$) temperature profile of 150°–220°C. The first type of maleated PEs was termed Reac-PE-*g*-MA. It was prepared by direct extrusion of MA, LLDPE and peroxide. The second type, PEMA–LLDPE, was prepared by melt blending with PEMA (Aldrich, 0.85 wt% MA grafted) and pure LLDPE. The reaction mixtures were premixed in a Henschel mixer before they were fed into the extruder.

2.4. Measurements

X-ray diffraction (XRD) was carried out by using Rigaku X-ray generator (Cu Kα radiation with $\lambda = 0.15406$ nm) at room temperature. The diffractograms were scanned in 2θ ranges from 1.2 to 10° at a rate of 2°/min. Transmission electron microscope, Pillips CM20, was used to observe the dispersibility of the clay in hybrids using an acceleration voltage of 120 kV. An ultrathin section of 70 nm in thickness was prepared by an ultramicrotome Leica EM FCS. Thermogravimetric analyzer (TGA) was used to observe the exchanged amount of alkylammonium to Na–MMT. Elemental analysis (EA) and FT-IR were used to measure the maleic anhydride (MA) grafting level to polyethylene prepared by melt extrusion. Elemental analysis was performed by CE EA-1110 Elemental Analyzer. The

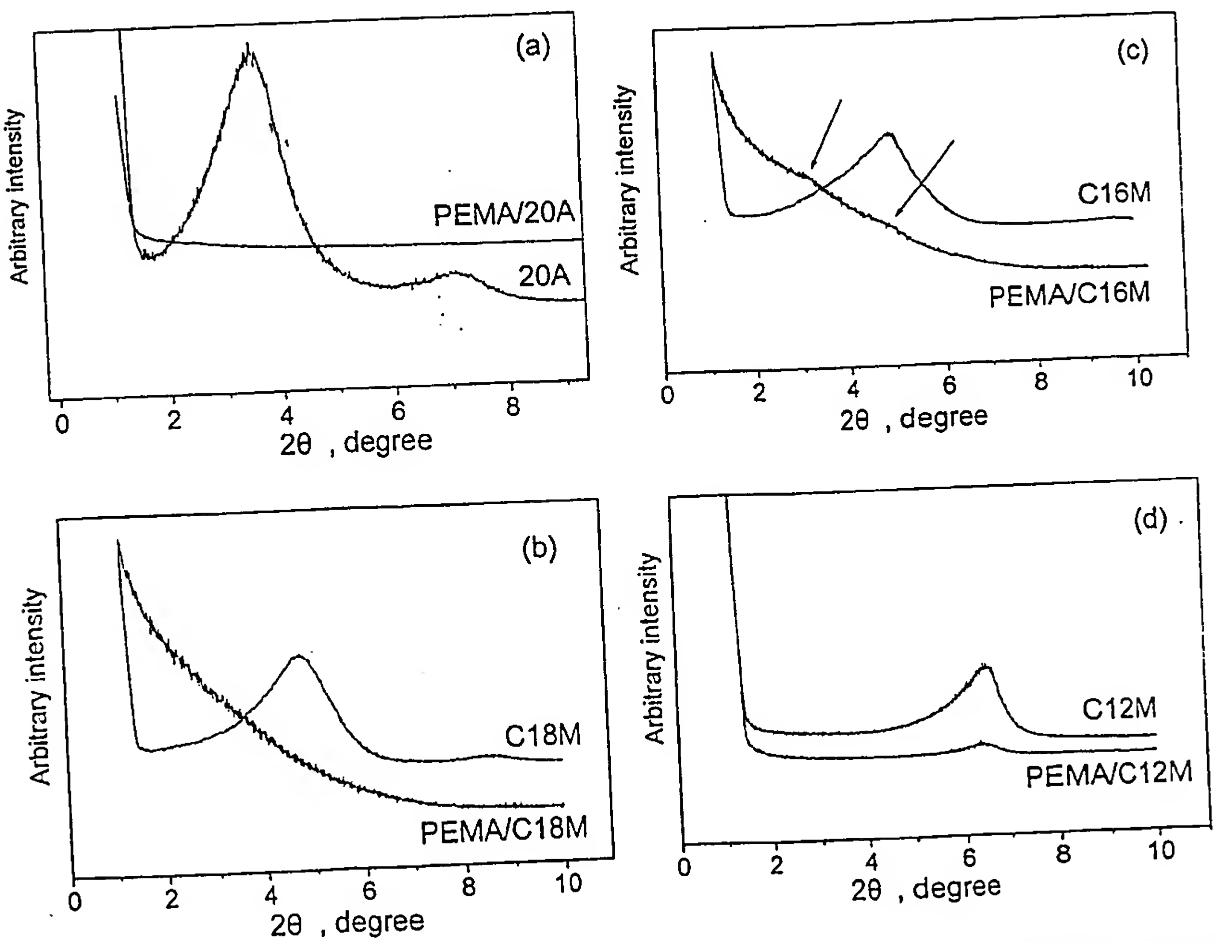

Fig. 3. XRD patterns of hybrids with PEMA and various organoclays (5 wt%): (a) 20A system, (b) C18M system, (c) C16M system, (d) C12M system.

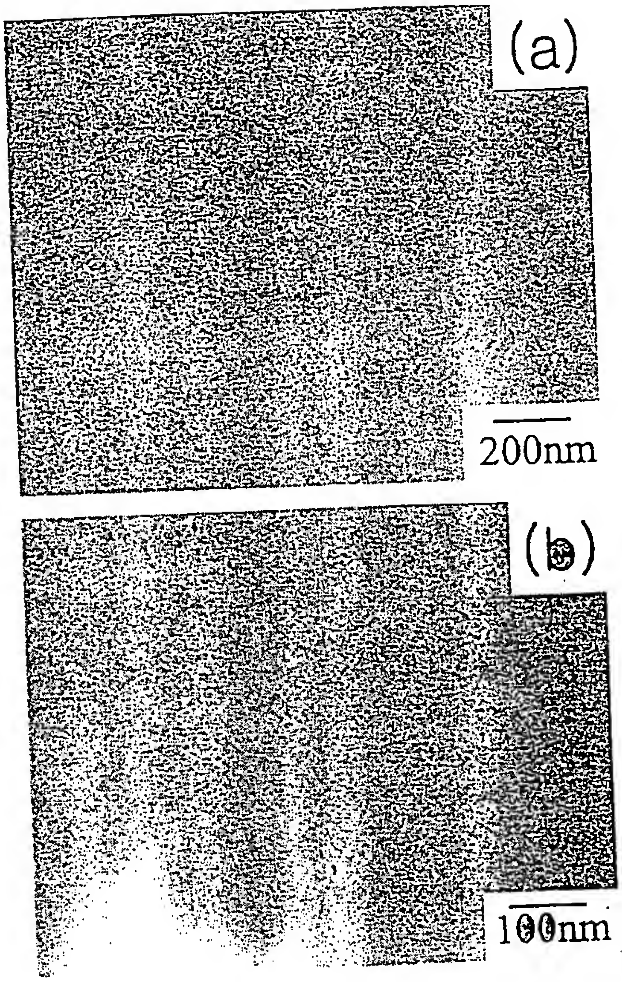

Fig. 4. (a) TEM of hybrid with 20A 5 wt%, (b) TEM of hybrid with C18M 5 wt%.

FT-IR spectra were recorded on a Bomen-MB-100 FT-IR spectrometer with a 4 cm^{-1} resolution. Molecular weight of polyethylene was measured by Gel Permeation Chromatography (GPC, Waters 150 CV) using Shodex linear HT800 column and 1,2,4-trichlorobenzene as eluent.

3. Results and discussion

3.1. Preparation of alkylamine modified MMT

The hydrophilic Na–montmorillonite (MMT) surface layers were modified by dispersing MMT uniformly into an organic modifier solution. As a result, the surface of the MMT had a hydrophobic character [37,38]. The hydrophobic organic modifier can facilitate the intercalation of a hydrophobic polymer into MMT by reducing the surface energy.

To investigate the effect of organic modifier on the morphology of polyethylene/clay nanocomposite, we prepared organoclays such as C12M, C16M and C18M with different alkylammonium chain lengths [4,22]. Also, an organophilic clay, 20A, had two long alkyl chains. Fig. 1 shows the thermo-gravimetric analysis (TGA) of modified clays. The TGA pattern of clays indicates that the clays are well ion exchanged with alkyl amines because the clay with the shorter chain length shows the smaller reduction in weight. Fig. 2 shows the X-ray diffraction patterns of organophilic clays. The interlayer distance is determined by the diffraction peak in the X-ray method, using the Bragg equation:

$$2d_{001}\sin\theta = \lambda$$

where d_{001} is the interplanar distance of (001) diffraction face, θ is the diffraction position and λ is the wavelength. The X-ray pattern shows clearly that the interlayer spacing increases with the increase in size of alkylamine chain length. The interlayer spacings of C12M, C16M, C18M and 20A are 1.36, 1.79, 1.85 and 2.47 nm, respectively. These spacings suggest the successful modification of MMT because the longer the chain length of the modifier becomes the larger the interlayer spacing.

3.2. Preparation of PEMA/clay nanocomposites by melt compounding

We prepared PEMA/clay nanocomposites by melt compounding and investigated the effect of organic modifier on the intercalation behavior of polymer. The MA grafting level of PEMA was 0.85 wt%. Fig. 3 shows the XRD curve of hybrids with PEMA and various modified MMTs. No basal peak reflections are shown for the hybrids with 20A and C18M in Fig. 3 (a) and (b) in the region from $2\theta = 2$ to 10°. This fact reveals that clays in both systems are exfoliated and dispersed homogeneously in the maleated polyethylene matrix. In order to confirm the nanoscale dispersion of clay, the morphology of hybrids with clay weight fraction of 5 wt% was observed by transmission electron microscopy (TEM). In Fig. 4 (a) and (b), the dark lines are the silicate layers. Each layer of clay is disordered and dispersed homogeneously in the PEMA. It is consistent with the absence of (001) plane peak in Fig. 3(a) and (b) due to very large interlayer spacing and the disordered state of silicate layer.

However, in the hybrid with C16M (Fig. 3 (c)), there is a weak peak at a lower angle ($2\theta = 3.2°$) than the basal reflection peak position of C16M ($2\theta = 4.9°$) and there is also a weak peak at the same angle of the modified clay. This indicates that some parts of C16M are intercalated by PEMA but some parts of C16M are not intercalated. Fig. 3(d) indicates that the peak position of original C12M ($2\theta = 6.5°$) is not changed and C12M is not intercalated by the maleated polyethylene. Apparently, a macrophase separation texture was observed (even though TEM is not shown here). It can be concluded that the hybrid with C12M is like a conventional composite.

From these results, it is found that the alkylammonium

Table 1
Grafted MA ratio (wt%) in Reac-PE-*g*-MA prepared by reactive extrusion using twin extruder

Blend ratio before reactive extrusion		Grafted MA wt% in Reac-PE-*g*-MA
LLDPE (wt%)	MA (wt%)	
99.9	0.1	0.068
99.8	0.2	0.11
99.7	0.3	0.22
99.6	0.4	0.29
99.5	0.5	0.33

chain length, i.e. hydrophobicity of organic modifier is an important factor to determine the morphology of PEMA/clay nanocomposite. Exfoliated or intercalated PEMA/clay nanocomposite can be obtained when the number of methylene units in alkylammonium chain is larger than 16.

3.3. Preparation of maleated PE/clay nanocomposite by melt compounding

In addition to the chain length of an organic modifier, the MA grafting level of PE may also be important to the morphology of polyethylene/clay nanocomposite. We prepared the maleated PE with a different MA grafting level by two methods as mentioned in Section 2: the reactive extrusion of LLDPE, MA and peroxide, and the melt blending of PEMA and LLDPE. In order to measure the content of MA grafted on LLDPE, the samples obtained after grafting reaction were pre-dried to remove the unreacted MA monomers in a vacuum oven at 60°C for 5 h. The dried samples were dissolved in xylene at a concentration of 2 wt%. This solution was mixed with acetone and the precipitate was filtered and dried again in a vacuum oven at 60°C for 24 h. The percentage of MA grafted on LLDPE was determined by elemental analysis (EA) and FT-IR spectra. The measured grafted levels of various types of the maleated PE were summarized in Tables 1 and 2.

Fig. 5(a) shows the FT-IR spectra of LLDPE, PEMA and

Table 2
Grafted MA ratio (wt%) in PEMA–LLDPE prepared by melt blend using single extruder (Aldrich PEMA: 0.85 wt% MA grafted, M_w = 126 000 by GPC)

Blend ratio before melt blend		Grafted MA wt% in PEMA–LLDPE
LLDPE (wt%)	PEMA[a] (wt%)	
100	0	0
95	5	0.04
90	10	0.09
80	20	0.17
70	30	0.26
50	50	0.43
0	100	0.85

[a] LLDPE:5MI, 0.930 density, M_w = 129 000 by GPC.

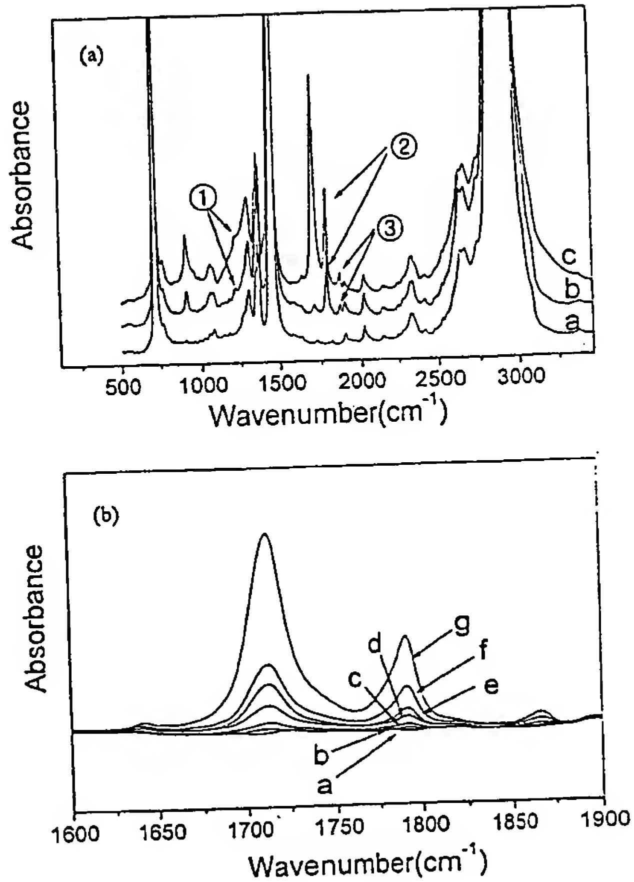

Fig. 5. (a) FT-IR curve of Reac-PE-*g*-MA obtained by reactive extrusion: a: pure LLDPE, b: MA contents: 0.33%, c: PEMA, (1) 1218 cm^{-1}, (2) 1791 cm^{-1}, (3) 1866 cm^{-1}; (b) FT-IR curve of PEMA–LLDPE obtained by melt blending of PEMA and LLDPE: MA contents; a: 0% (pure LLDPE), b: 0.04%, c: 0.09%, d: 0.17%, e: 0.26%, f: 0.43%, g: 0.85%.

maleated PE obtained by reactive extrusion. It is found that new peaks appear at 1218, 1791 and 1866 cm^{-1} in Reac-PE-*g*-MA when compared to the pure LLDPE. New peaks indicate that MA has been successfully grafted onto polyethylene chains [39,40]. Fig. 5(b) shows the increase of peak height at 1791 cm^{-1} with the increase of PEMA content. This peak increase is consistent with MA weight percentage, so the MA grafting level was measured from the intensity of 1791 cm^{-1}, and the thickness variation of each sample was corrected by 2019 cm^{-1} intensity for internal reference peak [41].

Maleated PE/clay nanocomposites are synthesized and the effect of grafting level of MA on their morphology is investigated. The contents of organically modified montmorillonite clay are fixed at 5 wt% in all hybrids. Fig. 6 shows the XRD patterns of hybrids with 20A. The original basal reflection peak of 20A disappears completely above a certain grafting level of MA, which is about 0.1 wt%. But below this critical grafting level of MA, a new basal reflection peak with very weak intensity appears

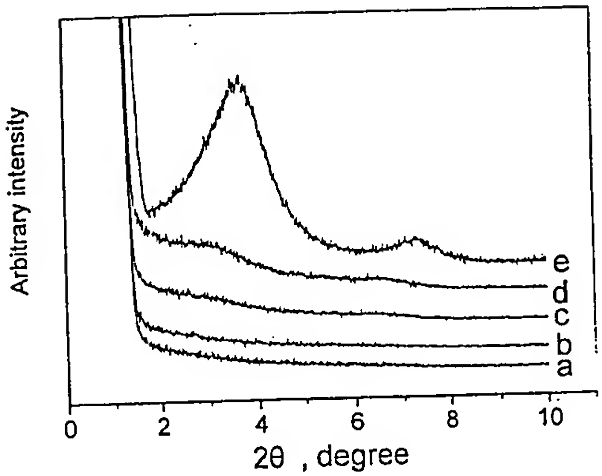

Fig. 6. XRD patterns of hybrids of 20A and Reac-PE-*g*-Mas with various MA grafting levels prepared by reactive extrusion: MA contents; (a) 0.29%, (b) 0.22%, (c) 0.11%, (d) 0.07%, (e) clay 20A only.

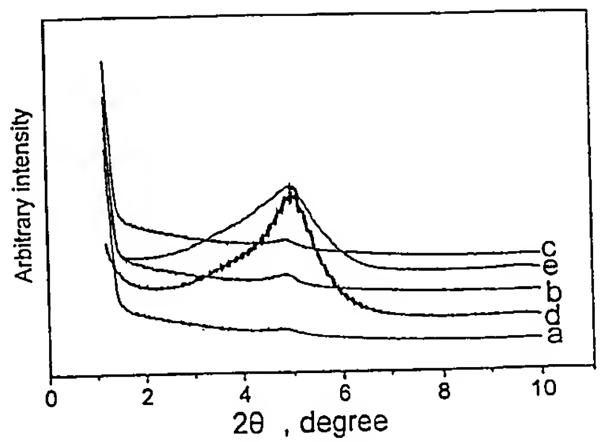

Fig. 8. XRD patterns for hybrids of C16M and Reac-PE-*g*-Mas with various MA grafting levels prepared by reactive extrusion: MA contents; (a) 0.33%, (b) 0.29%, (c) 0.22%, (d) 0% (pure LLDPE), (e) C16M only.

at lower angle than the peak of 20A and indicates the intercalation of some Reac-PE-*g*-MA into the clay layers.

Fig. 7 shows no distinct peak of clay C18M for all hybrids above 0.1 wt% of MA grafting level. This critical grafting level of MA is almost the same as the hybrid of Reac-PE-*g*-MA and 20A. In Fig. 8, the hybrids with C16M shows a weak peak at the same position of the original clay peak regardless of the grafting level. This indicates that most of clay is not dispersed into the matrix.

The hydrophobicity of the organically modified montmorillonite increases as the carbon number of organic modifier increases. On the other hand, the hydrophilicity of polyethylene increases as the grafting level of MA in polyethylene. This result suggests that in order to obtain good dispersion of clay in matrix, the polyethylene should have a certain grafting level of MA in matrix.

3.4. Intercalation behavior of pure LLDPE

The hybrid of pure LLDPE with C16M shows a clear peak at the same position as that of C16M in the XRD pattern (d curve in Fig. 8). This indicates no intercalation of LLDPE into C16M. On the other hands, the hybrids with 20A and C18M show peaks shifted toward lower angles than those of the clays (Figs. 7 and 9). This clearly indicates the strong intercalation of polymers. Therefore a proper modification of Na–MMT with dimethyl-dehydrogenated tallow (20A) and octadecylamine (C18M) may give an intercalated morphology in the pure LLDPE without grafting of maleic anhydride. Fig. 9 shows the intercalation

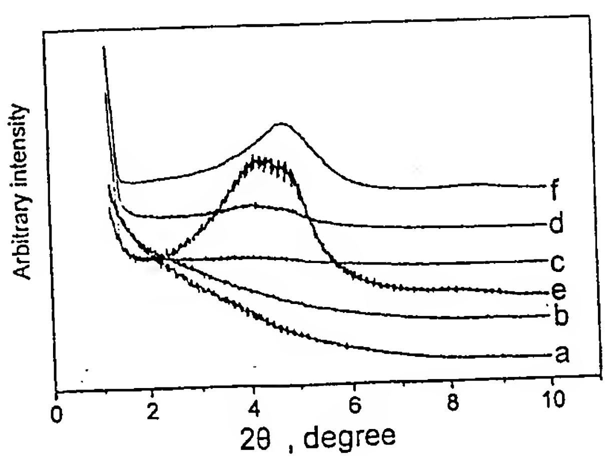

Fig. 7. XRD patterns for hybrids of C18M and PEMA–LLDPEs with various MA grafting levels prepared by melt blending: MA contents; (a) 0.85%, (b) 0.17%, (c) 0.09%, (d) 0.04%, (e) 0% (pure LLDPE), (f) C18M only.

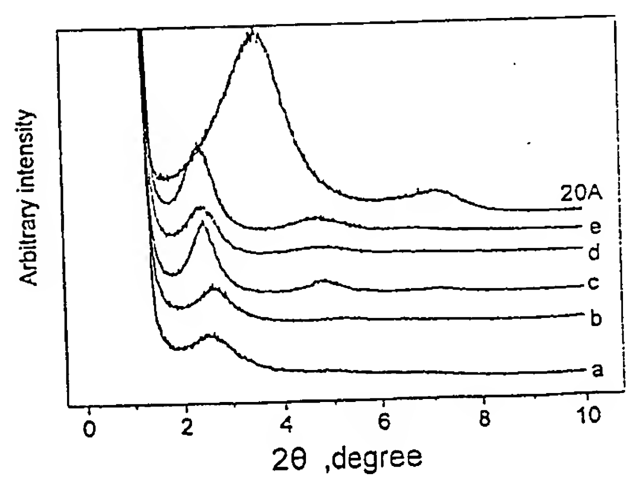

Fig. 9. XRD patterns for hybrids of pure LLDPE with various molecular weights and 20A: M_w of LLDPE by GPC; (a) 15 000, (b) 53 000, (c) 103 000, (d) 129 000, (e) 180 000.

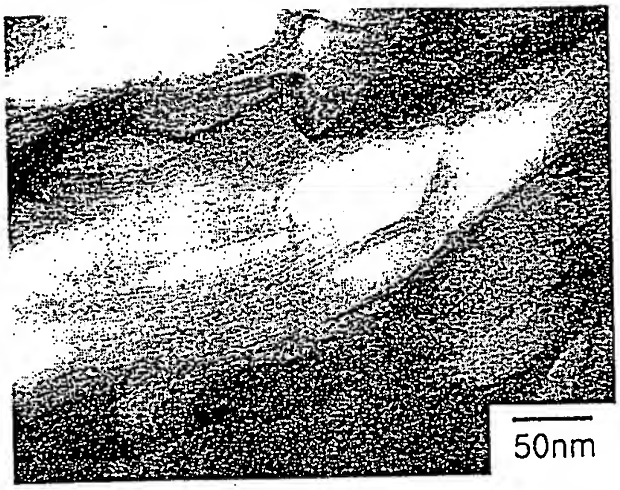

Fig. 10. TEM of hybrid with LLDPE (M_w: 129 000) and 5 wt% of 20A.

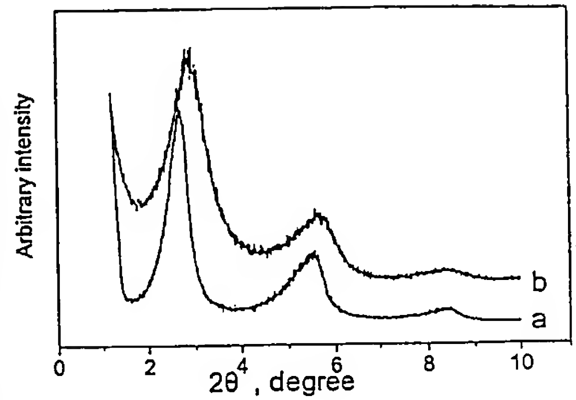

Fig. 11. XRD pattern of hybrid with increased interlayer C18M clay: (a) increased interlayer C18M (C18M(2)), (b) hybrid with pure LLDPE (M_w:129 000) and C18M(2).

behaviors of LLDPE with various molecular weights into 20A (5 wt%). The molecular weight of LLDPE does not significantly affect the morphology of the LLDPE/20A nanocomposite. Hence, it is hard to increase the interlayer spacing of clay by controlling the molecular weight of polyethylene. In order to confirm the dispersibility of 20A clay in pure LLDPE matrix, TEM was used as shown in Fig. 10. The interlayer spacing of clay is about 4 nm, which is almost coincident value expected by XRD peak (Fig. 9). It is realized that polyethylene nanocompoiste can be obtained by only proper modification of clay without modification of polyethylene and can be applied to many application areas with low fabrication cost.

This intercalation behavior of pure LLDPE may be originated from the initially higher interlayer spacing and the more compatible characteristics of the modifiers, 20A and C18M, with LLDPE than that of hexadecyl amine modifier, C16M.

In order to investigate the effect of interlayer spacing of modified clay, Na–MMT was modified with excess octadecyl amine. This new organophilic montmorillonite, C18M(2), has higher interlayer spacing (3.27 nm) than 1.85 nm of C18M used in the experiment (Fig. 11). But in the LLDPE/C18M(2) hybrid, the octadecylamines came out of the layers and the enlarged spacing between layers becomes narrower. This indicates that the intercalation capability has a certain limit. That is, it does not depend on the initial spacing but it seems to be affected by the thermodynamical equilibrium state of the hydrophilicity of clay surface, the hydrophobicity of a modifier and LLDPE.

4. Conclusion

It is quite reasonable to say that the hydrophobicity of the organically modified clay and the hydrophilicity of maleated polyethylene are very important factors to achieve the exfoliated and homogeneously dispersed clay nanocomposites.

When polyethylene has a higher grafting level of MA than the critical grafting level of MA (0.1 wt%) and the number of methylene groups in alkylamine chain has more than 16, polyethylene/clay nanocomposites are completely exfoliated. In the hybrids with pure LLDPE, the intercalation capability depends on the thermodynamical equilibrium state at the clay surface rather than the initial interlayer spacing of organophilic clay.

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Exhibit 2

Polymer
Volume 42, Issue 24 , November 2001, Pages 9819-9826

doi:10.1016/S0032-3861(01)00509-2 Cite or link using doi

Synthesis and characterization of maleated polyethylene/clay nanocomposites

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Abstract

Maleic anhydride grafted polyethylene (maleated polyethylene)/clay nanocomposites were prepared by simple melt compounding. The exfoliation and intercalation behaviors depended on the hydrophilicity of polyethylene grafted with maleic anhydride and the chain length of organic modifier in the clay. When the number of methylene groups in alkylamine (organic modifier) was larger than 16, the exfoliated nanocomposite was obtained, and the maleic anhydride grafting level was higher than about 0.1 wt% for the exfoliated nanocomposite with the clay modified with dimethyl dihydrogenated tallow ammonium ion or octadecylammonium ion. The pure LLDPE showed only the intercalation, which does not depend on the initial spacing between clay layers.

Author Keywords: Polyethylene; Nanocomposite; Melt compounding

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Polymer
Volume 42, Issue 24 , November 2001 , Pages 9819-9826

This Document
Abstract
Full Text + Links
PDF (329 K)

Actions
E-mail Article

Exhibit 3

Mehta, Sameer D.

From:	Mehta, Sameer D.
Sent:	Thursday, [redacted] 7:29 AM
To:	Riopell, Daniel J.
Subject:	HDPE Spreadsheet

Book2.xls

Nanoclay Formulations

Masterbatch for HDPE

TPO PP

Masterbatch	Resin	Nanoclay	MAH-g-PE	Coupling agent
Sample weight	Parts	Parts	Parts	Parts
MB-HDPE-1	M6020	Cloisite 15A	CMB24	A-1100
	25	50	25	0.52
MB-HDPE-2	MPP213	Cloisite 15A	CMB24	A-1100
	25	50	25	0.52
MB-HDPE-3	M6020	Cloisite 15A	AX8900	A-1100
	25	50	25	0.52
MB-HDPE-4	MPP213	Cloisite 15A	AX8900	A-1100
	25	50	25	0.52
MB-HDPE-5	M6020	Cloisite 15A	UE634	A-1100
	25	50	25	0.52
MB-HDPE-6	M6020	Cloisite 15A	UE648	A-1100
	25	50	25	0.52

Masterbatch let down in HDPE

Resin	Resin	Masterbatch	Total
Sample weight	Parts	Parts	
HDPE- Control 1	M6020	None	100
10 lb	100		
HDPE- Control 2	MPP213	None	
10 lb	100		100
HDPE-1	M6020	MB-HDPE-1	
10 lb	88	12	100
HDPE-2	MPP213	MB-HDPE-2	
10 lb	88	12	100

HDPE-3	M6020	MB-HDPE-3	
10 lb	88	12	100
HDPE-4	MPP213	MB-HDPE-4	
10 lb	88	12	100
HDPE-5	M6020	MB-HDPE-5	
10 lb	88	12	100
HDPE-6	M6020	MB-HDPE-6	
10 lb.	88	12	100

Physical PropertiesTo Be Measured on Film

MVTR, O2TR

Tensile, tear, dart drop

Flexural Modulus

MPP213 (0.55 MI, 0.958) is under G2008 in inventory

M6020 (2.0 MI, 0.960 density) in under G3707 in inventory

Cloisite 20A is under G4288 in inventory

UE634 (6 MI, 20% VA) is under G4256 in inventory

UE648 (0.45 MI, 19% VA) is under G2788 in inventory

Total
100.52
100.52
100.52
100.52
100.52
100.52

Exhibit 4

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 ~~2152~~ 254
Date: [redacted]
Time: [redacted]
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	170	210	220	220	230	230	230	220	190
Barrel	166	210	221	220	227	230	230	221	190
Melt	B.I.	210	B.I.	246	252	227	252	225	227

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**90**	%	Monomer Feed Tank	**46**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**46**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**290**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-11**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-Control-1
M6020
Hopper 2: 6
Vacuum installed but not running
Nitrogen off
Head Pressure: 300
RPM: 300

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 ~~216~~ 255
Date: [redacted]
Time:
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	210	240	250	250	260	260	260	250	200
Barrel	187	241	250	250	260	260	260	250	200
Melt	B.I.	251	B.I.	287	286	264	284	251	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**92**	%	Monomer Feed Tank	**47**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**47**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**260**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-12**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-Control-2
MPP213
Hopper 2: 3.5
Vacuum installed but not running
Nitrogen off
Head Pressure: 270
RPM: 300

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30-~~219~~258
Date: [redacted]
Time: [redacted]
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	200	220	230	230	240	240	240	230	190
Barrel	173	220	231	230	239	240	240	229	189
Melt	B.I.	218	B.I.	259	274	240	261	234	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**85**	%	Monomer Feed Tank	**44**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**44**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**263**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-9**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-1
88% M6020, 12% MB-HDPE-1
Hopper 2: 6.0
Vacuum installed but not running
Nitrogen off
Head Pressure: 250
RPM: 300
Zone 8 not controlling correctly.
Zone 6 Barrel Temp reads the same as Set Point (bad input?)

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30-~~217~~256
Date: [redacted]
Time:
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	210	240	250	250	260	260	260	255	190
Barrel	197	240	250	250	258	260	259	256	189
Melt	B.I.	255	B.I.	289	292	263	291	255	B.I.

Print

--------------------Extruder Conditions--------------------

Screw Speed	B.I.	rpm	Monnomer Pump Speed	500	%
Extr Torque	91	%	Monomer Feed Tank	47	C
Kilowatts	10.0	KW	Monomer Feed Rate	47	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	319	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-11	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-2
88% MPP213, 12% MB-HDPE-2
Hopper 2: 3.0
Vacuum installed but not running
Nitrogen off
Head Pressure: 350
RPM: 300

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30-~~220~~ 259
Date: [redacted]
Time: [redacted]
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	200	220	230	230	240	240	240	230	190
Barrel	172	219	229	230	240	240	240	248	191
Melt	B.I.	217	B.I.	260	271	242	275	254	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**87**	%	Monomer Feed Tank	**44**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**44**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**265**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-11**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-3
88% M6020, 12% MB-HDPE-3
Hopper 2: 5.6
Vacuum installed but not running
Nitrogen off
Head Pressure: 270
RPM: 300
Zone 8 not controlling correctly.
Zone 6 Barrel Temp reads the same as Set Point (bad input?)

Equistar Chemicals LP **ETC CPMU ZSK-30 RPD**

Run No.: CPMU 30-~~218~~257
Date: [redacted]
Time: [redacted]
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	210	240	250	250	260	260	260	255	190
Barrel	198	240	251	250	257	260	260	212	189
Melt	B.I.	258	B.I.	297	332	265	288	212	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	B.I.	rpm	Monnomer Pump Speed	500	%
Extr Torque	95	%	Monomer Feed Tank	49	C
Kilowatts	10.0	KW	Monomer Feed Rate	49	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	309	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-5	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-4
88% MPP213, 12% MB-HDPE-4
Hopper 2: 2.6
Vacuum installed but not running
Nitrogen off
Head Pressure: 310
RPM: 300

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.: CPMU 30-~~221~~ 260
Date: [redacted]
Time: [redacted]
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	200	220	230	230	240	240	240	225	190
Barrel	172	220	229	229	240	240	240	222	190
Melt	B.I.	214	B.I.	259	268	241	256	227	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**79**	%	Monomer Feed Tank	**40**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**40**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**270**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-12**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-5
88% M6020, 12% MB-HDPE-5
Hopper 2: 6.5
Vacuum installed but not running
Nitrogen off
Head Pressure: 280
RPM: 300
Zone 8 not controlling correctly.
Zone 6 Barrel Temp reads the same as Set Point (bad input?)

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30-~~222~~26/
Date: [redacted]
Time:
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	200	220	230	230	240	240	240	225	190
Barrel	172	219	231	229	241	240	241	224	190
Melt	B.I.	213	B.I.	260	269	241	258	230	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	B.I.	rpm	Monnomer Pump Speed	500	%
Extr Torque	80	%	Monomer Feed Tank	41	C
Kilowatts	10.0	KW	Monomer Feed Rate	41	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	275	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-16	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

HDPE-6
88% M6020, 12% MB-HDPE-6
Hopper 2: 6.5
Vacuum installed but not running
Nitrogen off
Head Pressure: 300
RPM: 300
Zone 8 not controlling correctly.
Zone 6 Barrel Temp reads the same as Set Point (bad input?)

Exhibit 5

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11160
Page 1

[redacted] 09:39 by SDMEHTA

Sample : A00-11160
Sample Status : A
Login by : SDM005
Date Logged in: [redacted]
9:41:54
Auth/X by : BATCH
Date Auth/X : [redacted]
3:02:43
Submitter : SDM005
Project Number: PPROP-004

Q Prod. Name :
Lot Number :

Customer Name :
Sample Type : HDPE

Sample Form : PELLETS
Notebook # :

Reason Code : PROD_DEV
Sample Name : HDPE-Control
1
Job Name : SDM_NANO_HDPE_TEST
Hazard : 0-1-0
Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

These results are subject to verification and approval.

Analysis		R	Component Name	Result	St
AASH	/1	N	Ash content	80 PPM	A
		T	Notebook	WXW-1-16	A
ADORS	/1	T	RDR	2.46	A
		N	a value	5.0	A
		N	ab value	0.72	A
		N	% error	1.26	A
		N	Test Temp	190 deg_C	A
		N	% Strain	20	A

	N	Plate Size	25 mm	A
	N	ETA*100	10600.0 poise	A
	T	ER	4.15	A
	T	PDR	21.66	A
	T	ET	NR	A
	T	YR	2127	A
	N	Slope (ETA-0,GAMMA DOT-0	1.00	A
	T	Run #	HD0500	A
	T	Comment	PAH082400	A
AFR1902160/1	N	Flow rate	0.918 Grams/10 m	A
	T	Notebook	DEE-1	A

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11162
Page 1

[redacted] 09:40 by SDMEHTA

Sample : A00-11162
Login by : SDM005
9:42:59
Auth/X by : BATCH
3:02:44
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV
2
Job Name : SDM_NANO_HDPE_TEST
Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-Control

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	130 PPM
	A	T	Notebook	WXW-1-16
ADORS /1	A	T	RDR	.46
	A	N	a value	4.0
	A	N	ab value	0.72
	A	N	% error	1.29
	A	N	Test Temp	190 deg_C
	A	N	% Strain	20

```
|              |N | Plate Size                  | 25 mm
      |A |
|              |N | ETA*100                     | 14000.0 poise
      |A |
|              |T | ER                          | 2.23
      |A |
|              |T | PDR                         | 7.13
      |A |
|              |T | ET                          | NR
      |A |
|              |T | YR                          | 1382
      |A |
|              |N | Slope (ETA-0,GAMMA DOT-0    | 1.00
      |A |
|              |T | Run #                       | HD0500
      |A |
|              |T | Comment                     | PAH082400
      |A |
|AFR1902160/1  |N | Flow rate                   | 1.17 Grams/10 m
      |A |
|              |T | Notebook                    | DEE-1
      |A |
----------------------------------------------------------------------
----------
```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11164
Page 1

[redacted] 09:41 by SDMEHTA

Sample : A00-11164
Login by : SDM005
9:44:23
Auth/X by : BATCH
3:02:50
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_TEST
Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-1

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	33670 PPM
	A	T	Notebook	WXW-1-16
ADORS /1	A	T	RDR	1.58
	A	N	a value	3.3
	A	N	ab value	0.72
	A	N	% error	0.91
	A	N	Test Temp	190 deg_C
	A	N	% Strain	20

```
|              |N | Plate Size                  | 25 mm
       |A |
|              |N | ETA*100                     | 10700.0 poise
       |A |
|              |T | ER                          | 2.02
       |A |
|              |T | PDR                         | 9.66
       |A |
|              |T | ET                          | NR
       |A |
|              |T | YR                          | 1294
       |A |
|              |N | Slope (ETA-0,GAMMA DOT-0    | 1.00
       |A |
|              |T | Run #                       | HD0500
       |A |
|              |T | Comment                     | PAH082400
       |A |
|AFR1902160/1  |N | Flow rate                   | 1.48 Grams/10 m
       |A |
|              |T | Notebook                    | DEE-1
       |A |
----------------------------------------------------------------------
----------
```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11166
Page 1

[redacted] 09:42 by SDMEHTA

Sample : A00-11166
Login by : SDM005
9:45:08
Auth/X by : BATCH
3:02:50
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_TEST
Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-2

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis		R	Component Name	Result	St
AASH	/1	N	Ash content	33190 PPM	A
		T	Notebook	WXW-1-16	A
ADORS	/1	T	RDR	.75	A
		N	a value	2.6	A
		N	ab value	0.72	A
		N	% error	1.98	A
		N	Test Temp	190 deg_C	A
		N	% Strain	20	A

	N	Plate Size	25 mm	A
	N	ETA*100	20800.0 poise	A
	T	ER	1.21	A
	T	PDR	3.88	A
	T	ET	NR	A
	T	YR	360.4	A
	N	Slope (ETA-0,GAMMA DOT-0	1.00	A
	T	Run #	HD0500	A
	T	Comment	PAH082400	A
AFR1902160/1	N	Flow rate	0.821 Grams/10 m	A
	T	Notebook	DEE-1	A

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11167
Page 1

[redacted] 09:42 by SDMEHTA

Sample : A00-11167
Login by : SDM005
9:45:40
Auth/X by : BATCH
3:02:50
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_TEST

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-3

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	32850 PPM
	A	T	Notebook	WXW-1-16
ADORS /1	A	T	RDR	1.87
	A	N	a value	3.0
	A	N	ab value	0.72
	A	N	% error	0.62
	A	N	Test Temp	190 deg_C
	A	N	% Strain	20

```
|              |N | Plate Size                | 25 mm
       |A |
|              |N | ETA*100                   | 9780.0 poise
       |A |
|              |T | ER                        | 1.92
       |A |
|              |T | PDR                       | 9.06
       |A |
|              |T | ET                        | NR
       |A |
|              |T | YR                        | 793.8
       |A |
|              |N | Slope (ETA-0,GAMMA DOT-0  | 1.00
       |A |
|              |T | Run #                     | HD0500
       |A |
|              |T | Comment                   | PAH082400
       |A |
|AFR1902160/1  |N | Flow rate                 | 1.465 Grams/10 m
       |A |
|              |T | Notebook                  | DEE-1
       |A |
----------------------------------------------------------------------
----------
```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

[redacted] 09:42 by SDMEHTA

Sample : A00-11168
Login by : SDM005
9:46:12
Auth/X by : BATCH
3:02:51
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_TEST
Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-4

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis		St	R	Component Name	Result
AASH	/1	A	N	Ash content	36820 PPM
		A	T	Notebook	WXW-1-16
ADORS	/1	A	T	RDR	.82
		A	N	a value	2.4
		A	N	ab value	0.72
		A	N	% error	1.76
		A	N	Test Temp	190 deg_C
		A	N	% Strain	20

	A	N	Plate Size	25 mm
	A	N	ETA*100	21100.0 poise
	A	T	ER	1.10
	A	T	PDR	3.45
	A	T	ET	NR
	A	T	YR	470.5
	A	N	Slope (ETA-0,GAMMA DOT-0	1.00
	A	T	Run #	HD0500
	A	T	Comment	PAH082400
AFR1902160/1	A	N	Flow rate	0.815 Grams/10 m
	A	T	Notebook	DEE-1

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11169
Page 1

[redacted] 09:43 by SDMEHTA

Sample : A00-11169
Login by : SDM005
9:46:42
Auth/X by : BATCH
3:02:51
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_TEST

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-5

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

These results are subject to verification and approval.

Analysis		R	Component Name	Result	St
AASH	/1	N	Ash content	32100 PPM	A
		T	Notebook	WXW-1-16	A
ADORS	/1	T	RDR	1.78	A
		N	a value	3.6	A
		N	ab value	0.72	A
		N	% error	0.96	A
		N	Test Temp	190 deg_C	A
		N	% Strain	20	A

```
|              |N | Plate Size                   | 25 mm
        |A |
|              |N | ETA*100                      | 10200.0 poise
        |A |
|              |T | ER                           | 2.69
        |A |
|              |T | PDR                          | 11.96
        |A |
|              |T | ET                           | NR
        |A |
|              |T | YR                           | 1722
        |A |
|              |N | Slope (ETA-0,GAMMA DOT-0     | 1.00
        |A |
|              |T | Run #                        | HD0500
        |A |
|              |T | Comment                      | PAH082400
        |A |
|AFR1902160/1  |N | Flow rate                    | 1.41 Grams/10 m
        |A |
|              |T | Notebook                     | DEE-1
        |A |
----------------------------------------------------------------------
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```

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-11170
Page 1

[redacted] 09:43 by SDMEHTA

Sample	: A00-11170	Sample Status	: A
Login by	: SDM005	Date Logged in:	[redacted]
9:47:12			
Auth/X by	: BATCH	Date Auth/X	: [redacted]
3:02:46			
Submitter	: SDM005	Project Number:	PPROP-004
Q Prod. Name	:	Lot Number	:
Customer Name	:	Sample Type	: HDPE
Sample Form	: PELLETS	Notebook #	:
Reason Code	: PROD_DEV	Sample Name	: HDPE-6
Job Name	: SDM_NANO_HDPE_TEST	Hazard	: 0-1-0

Description of Problem : (SEE BELOW)
Please run DORS (Y-value)

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	33500 PPM
	A	T	Notebook	WXW-1-16
ADORS /1	A	T	RDR	2.00
	A	N	a value	3.0
	A	N	ab value	0.72
	A	N	% error	0.68
	A	N	Test Temp	190 deg_C
	A	N	% Strain	20

	N	Plate Size	25 mm	A
	N	ETA*100	10000.0 poise	A
	T	ER	2.18	A
	T	PDR	9.57	A
	T	ET	NR	A
	T	YR	961.9	A
	N	Slope (ETA-0,GAMMA DOT-0	1.00	A
	T	Run #	HD0500	A
	T	Comment	PAH082400	A
AFR1902160/1	N	Flow rate	1.53 Grams/10 m	A
	T	Notebook	DEE-1	A

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

RESULT LIST

Exhibit 6

Masterbatch let down in HDPE

Resin Sample weight	Resin Parts	Masterbatch Parts	Total
HDPE-C1-ZSK30	M6020	None	100
10 lb	100		
HDPE-C2-ZSK30	MPP213	None	
10 lb	100		100
HDPE-C1-commercial	M6020	None	100
10 lb	100		
HDPE-C2-commercial	MPP213	None	
10 lb	100		100
HDPE-7	M6020	MB-HDPE-1	
10 lb	88	12	100
HDPE-8	MPP213	MB-HDPE-2	
10 lb	88	12	100
HDPE-9	M6020	MB-HDPE-3	
10 lb	88	12	100
HDPE-10	MPP213	MB-HDPE-4	
10 lb	88	12	100
HDPE-11	M6020	MB-HDPE-5	
10 lb	88	12	100
HDPE-12	M6020	MB-HDPE-6	
10 lb.	88	12	100
HDPE-13	GATR311	MB-HDPE-1	
10 lb	88	12	100
HDPE-14	L4907	MB-HDPE-1	
10 lb.	88	12	100

Physical PropertiesTo Be Measured on Film

MVTR, O2TR

Tensile, tear, dart drop, puncture, winzen

Flexural Modulus

MPP213 (0.55 MI, 0.958) is under G2008 in inventory

M6020 (2.0 MI, 0.960 density) in under G3707 in inventory VTA9121703

Cloisite 20A is under G4288 in inventory

UE634 (6 MI, 20% VA) is under G4256 in inventory LP291115F1

UE648 (0.45 MI, 19% VA) is under G2788 in inventory LP290624H2

Same as HDPE 1-6 except that the let down are done on a less harsh screw on ZSK-30

GATR311 is under G5307 in inventory

L4907 is under G1724 in inventory MT29040521

Exhibit 7

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 264
Date: [redacted]
Time: 1:34:15
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	170	200	210	210	215	220	219	210	199
Melt	B.I.	188	B.I.	187	182	219	236	221	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	B.I.	rpm	Monnomer Pump Speed	500	%
Extr Torque	48	%	Monomer Feed Tank	20	C
Kilowatts	10.0	KW	Monomer Feed Rate	20	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	358	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-10	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments:

HDPE-Control-1 soft screw
M6020
Hopper 2: 12
Nitrogen off
Head Pressure: 380
RPM: 250

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 265
Date: [redacted]
Time: 3:47:33
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	177	200	209	210	222	220	219	210	197
Melt	B.I.	190	B.I.	190	190	217	237	220	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	B.I.	rpm	Monnomer Pump Speed	500	%
Extr Torque	53	%	Monomer Feed Tank	22	C
Kilowatts	10.0	KW	Monomer Feed Rate	22	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	698	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-13	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments:

HDPE-Control-2 soft screw
MPP213
Hopper 2: 12
Nitrogen off
Head Pressure: 750
RPM: 250

Equistar Chemicals LP **ETC CPMU ZSK-30 RPD**

Run No.: CPMU 30 266
Date: [redacted]
Time: 9:09:29
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	167	200	210	211	205	220	224	210	200
Melt	B.I.	182	B.I.	183	173	221	232	219	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	238	rpm	Monnomer Pump Speed	500	%
Extr Torque	50	%	Monomer Feed Tank	21	C
Kilowatts	10.0	KW	Monomer Feed Rate	21	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	396	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-18	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-7

HDPE-1 with soft screw
88% M6020, 12% MB-HDPE-1
Hopper 2: 15.0
Nitrogen off
Head Pressure: 410
RPM: 250

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 270
Date: [redacted]
Time: 10:48:14
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	220	220
Barrel	169	200	211	209	215	220	218	223	210
Melt	B.I.	183	B.I.	178	182	218	232	234	B.I.

Print

--------------------Extruder Conditions--------------------

Screw Speed	238	rpm	Monnomer Pump Speed	500	%
Extr Torque	59	%	Monomer Feed Tank	24	C
Kilowatts	10.0	KW	Monomer Feed Rate	24	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	675	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-3	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-8

HDPE-2 with soft screw
88% Mpp213, 12% MB-HDPE-2
Hopper 2: 15.0
Nitrogen off
Head Pressure: 750
RPM: 250
Severe melt fracture

Equistar Chemicals LP ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 2697
Date: [redacted]
Time: 9:58:47
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	167	200	211	211	213	220	220	210	200
Melt	B.I.	179	B.I.	169	182	221	231	220	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	238	rpm	Monnomer Pump Speed	500	%
Extr Torque	47	%	Monomer Feed Tank	19	C
Kilowatts	10.0	KW	Monomer Feed Rate	19	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	364	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-8	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-9

HDPE-3 with soft screw
88% M6020, 12% MB-HDPE-3
Hopper 2: 15.0
Nitrogen off
Head Pressure: 380
RPM: 250

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.: CPMU 30 271
Date: [redacted]
Time: 12:47:23
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	220	220
Barrel	174	199	208	209	218	220	218	220	219
Melt	B.I.	186	B.I.	183	183	218	234	231	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**B.I.**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**62**	%	Monomer Feed Tank	**26**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**26**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**728**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-9**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-16

HDPE-4 with soft screw
88% Mpp213, 12% MB-HDPE-4
Hopper 2: 15.0
Nitrogen off
Head Pressure: 740
RPM: 250
Severe melt fracture

Equistar Chemicals LP   ETC CPMU ZSK-30 RPD

Run No.:	CPMU 30 268
Date:	[redacted]
Time:	9:58:19
Resin Type:	Compound Blend
Monomers Used:	Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	167	200	211	211	213	220	221	210	200
Melt	B.I.	179	B.I.	169	182	222	231	219	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	**238**	rpm	Monnomer Pump Speed	**500**	%
Extr Torque	**47**	%	Monomer Feed Tank	**19**	C
Kilowatts	**10.0**	KW	Monomer Feed Rate	**19**	pph
Vacuum	**500**	mmHg	Mon Zone 1		LBS.
Head Pressure	**368**	psi	Mon Zone 2	**19.00**	LBS.
Barrel Pressure	**-8**	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-17

HDPE-5 with soft screw
88% M6020, 12% MB-HDPE-5
Hopper 2: 15.0
Nitrogen off
Head Pressure: 380
RPM: 250

Equistar Chemicals LP **ETC CPMU ZSK-30 RPD**

Run No.: CPMU 30 269
Date: [redacted]
Time: 10:19:29
Resin Type: Compound Blend
Monomers Used: Nano Composite Master Batch Letdown in HDPE

	Z1	Z2	Z3	Z4	Z5	Z6	Z7	Z8	Z9
Setpoint	190	200	210	210	220	220	220	210	200
Barrel	168	200	210	210	215	220	218	210	201
Melt	B.I.	176	B.I.	171	184	217	228	219	B.I.

Print

------------------Extruder Conditions------------------

Screw Speed	238	rpm	Monnomer Pump Speed	500	%
Extr Torque	47	%	Monomer Feed Tank	20	C
Kilowatts	10.0	KW	Monomer Feed Rate	20	pph
Vacuum	500	mmHg	Mon Zone 1		LBS.
Head Pressure	364	psi	Mon Zone 2	19.00	LBS.
Barrel Pressure	-9	psi	Mon Zone 3		LBS.

Coordinators: Dan Riopell

Comments: HDPE-12

HDPE-6 with soft screw
88% M6020, 12% MB-HDPE-6
Hopper 2: 15.0
Nitrogen off
Head Pressure: 400
RPM: 250

Exhibit 8

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13028
Page 1

[redacted] at 07:53 by SDMEHTA

Sample : A00-13028
Login by : SDM005
3:57:16
Auth/X by : BATCH
3:03:50
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-7

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	32790 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9730 Grams/cc
	A	T	Notebook	wxw-2-40
ADORS /1	A	T	RDR	NR - Error too high.
	A	N	a value	5.0
		N	ab value	0.72

```
        |A |
|             |N | % error                    | 3.71
        |A |
|             |N | Test Temp                  | 190 deg_C
        |A |
|             |N | % Strain                   | 20
        |A |
|             |N | Plate Size                 | 25 mm
        |A |
|             |N | ETA*100                    | 11700.0 poise
        |A |
|             |T | ER                         | NR - Cor. coeff. too low
        |A |
|             |T | PDR                        | 13.28
        |A |
|             |T | ET                         | NR
        |A |
|             |T | YR                         | 1938
        |A |
|             |N | Slope (ETA-0,GAMMA DOT-0 | 1.00
        |A |
|             |T | Run #                      | HDPE1000
        |A |
|             |T | Comment                    | PAH100300
        |A |
|AFR1902160/1 |N | Flow rate                  | 1.46 Grams/10 m
        |A |
|             |T | Notebook                   | BRH4-136
        |A |
|AFR190HL   /1 |N | Flow rate                 | 77.3 Grams/10 m
        |A |
|             |T | Notebook                   | BRH4-134
        |A |
|AGPC       /1 |T | Comment                   | results sent to submitte
r       |A |
|AGPC       /2 |T | Comment                   | results sent to submitte
r       |A |
----------------------------------------------------------------------
----------
```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13029
Page 1

[redacted] at 07:55 by SDMEHTA

Sample : A00-13029
Login by : SDM005
3:57:44
Auth/X by : BATCH
3:03:51
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-8

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	R	St	Component Name	Result
AASH /1	N	A	Ash content	33460 PPM
	T	A	Notebook	WXW-2-35
ADENSANEX /1	N	A	Average density	0.9702 Grams/cc
	T	A	Notebook	wxw-2-40
ADORS /1	T	A	RDR	NR - Error too high.
	N	A	a value	3.0
	N		ab value	0.72

```
        |A |
|                 |N | % error                      | 4.17
        |A |
|                 |N | Test Temp                    | 190 deg_C
        |A |
|                 |N | % Strain                     | 20
        |A |
|                 |N | Plate Size                   | 25 mm
        |A |
|                 |N | ETA*100                      | 25700.0 poise
        |A |
|                 |T | ER                           | NR - Cor. coeff. too low
.       |A |
|                 |T | PDR                          | 5.14
        |A |
|                 |T | ET                           | NR
        |A |
|                 |T | YR                           | 1203
        |A |
|                 |N | Slope (ETA-0,GAMMA DOT-0     | 1.00
        |A |
|                 |T | Run #                        | HDPE1000
        |A |
|                 |T | Comment                      | PAH100300
        |A |
|AFR1902160/1     |N | Flow rate                    | 0.500 Grams/10 m
        |A |
|                 |T | Notebook                     | BRH4-136
        |A |
|AFR190HL   /1    |N | Flow rate                    | 15.8 Grams/10 m
        |A |
|                 |T | Notebook                     | BRH4-135
        |A |
|AGPC       /1    |T | Comment                      | results sent to submitte
r       |A |
|AGPC       /2    |T | Comment                      | results sent to submitte
r       |A |
--------------------------------------------------------------------
----------

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.
```

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13030
Page 1

[redacted] at 07:53 by SDMEHTA

Sample : A00-13030
Login by : SDM005
3:58:07
Auth/X by : BATCH
3:03:51
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-9

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	36900 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9776 Grams/cc
	A	T	Notebook	wxw-2-40
ADORS /1	A	T	RDR	2.14
	A	N	a value	2.6
		N	ab value	0.72

```
            |A  |
|                   |N  | % error                       | 1.50
            |A  |
|                   |N  | Test Temp                     | 190 deg_C
            |A  |
|                   |N  | % Strain                      | 20
            |A  |
|                   |N  | Plate Size                    | 25 mm
            |A  |
|                   |N  | ETA*100                       | 10900.0 poise
            |A  |
|                   |T  | ER                            | 1.67
            |A  |
|                   |T  | PDR                           | 7.65
            |A  |
|                   |T  | ET                            | NR
            |A  |
|                   |T  | YR                            | 657.4
            |A  |
|                   |N  | Slope (ETA-0,GAMMA DOT-0      | 1.00
            |A  |
|                   |T  | Run #                         | HDPE1000
            |A  |
|                   |T  | Comment                       | PAH100300
            |A  |
|AFR1902160/1       |N  | Flow rate                     | 1.57 Grams/10 m
            |A  |
|                   |T  | Notebook                      | BRH4-137
            |A  |
|AFR190HL   /1      |N  | Flow rate                     | 74.3 Grams/10 m
            |A  |
|                   |T  | Notebook                      | BRH4-138
            |A  |
|AGPC       /1      |T  | Comment                       | results sent to submitte
r           |A  |
|AGPC       /2      |T  | Comment                       | results sent to submitte
r           |A  |
----------------------------------------------------------------------
----------

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.
```

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13031
Page 1

[redacted] at 07:55 by SDMEHTA

Sample : A00-13031
Login by : SDM005
3:58:31
Auth/X by : BATCH
3:03:52
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-10

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	33780 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9704 Grams/cc
	A	T	Notebook	wxw-2-40
ADORS /1	A	T	RDR	.96
	A	N	a value	2.3
		N	ab value	0.72

```
        |A |
|                |N | % error                    | 1.73
        |A |
|                |N | Test Temp                  | 190 deg_C
        |A |
|                |N | % Strain                   | 20
        |A |
|                |N | Plate Size                 | 25 mm
        |A |
|                |N | ETA*100                    | 25300.0 poise
        |A |
|                |T | ER                         | 1.05
        |A |
|                |T | PDR                        | 3.40
        |A |
|                |T | ET                         | NR
        |A |
|                |T | YR                         | 344.8
        |A |
|                |N | Slope (ETA-0,GAMMA DOT-0   | 1.00
        |A |
|                |T | Run #                      | HDPE1000
        |A |
|                |T | Comment                    | PAH100300
        |A |
|AFR1902160/1    |N | Flow rate                  | 0.594 Grams/10 m
        |A |
|                |T | Notebook                   | BRH4-139
        |A |
|AFR190HL   /1   |N | Flow rate                  | 16.1 Grams/10 m
        |A |
|                |T | Notebook                   | BRH4-138
        |A |
|AGPC       /1   |T | Comment                    | results sent to submitte
r       |A |
|AGPC       /2   |T | Comment                    | results sent to submitte
r       |A |
----------------------------------------------------------------------
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```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

[redacted] at 07:54 by SDMEHTA

Sample : A00-13032
Login by : SDM005
4:16:51
Auth/X by : BATCH
3:03:52
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-11

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	31350 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9699 Grams/cc
	A	T	Notebook	wxw-2-40
ADORS /1	A	T	RDR	1.02
	A	N	a value	4.8
		N	ab value	0.72

```
        |A |
|            |N | % error                        | 2.18
        |A |
|            |N | Test Temp                      | 190 deg_C
        |A |
|            |N | % Strain                       | 20
        |A |
|            |N | Plate Size                     | 25 mm
        |A |
|            |N | ETA*100                        | 10800.0 poise
        |A |
|            |T | ER                             | NR - Cor. coeff. too low
        |A |
|            |T | PDR                            | 13.28
        |A |
|            |T | ET                             | NR
        |A |
|            |T | YR                             | 1950
        |A |
|            |N | Slope (ETA-0,GAMMA DOT-0       | 1.00
        |A |
|            |T | Run #                          | HDPE1000
        |A |
|            |T | Comment                        | PAH100300
        |A |
|AFR1902160/1 |N | Flow rate                      | 1.66 Grams/10 m
        |A |
|            |T | Notebook                       | BRH4-139
        |A |
|AFR190HL  /1 |N | Flow rate                      | 100 Grams/10 m
        |A |
|            |T | Notebook                       | BRH4-138
        |A |
|AGPC      /1 |T | Comment                        | results sent to submitte
r       |A |
|AGPC      /2 |T | Comment                        | results sent to submitte
r       |A |
----------------------------------------------------------------------
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Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.
```

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13033
Page 1

[redacted] at 07:54 by SDMEHTA

```
Sample          : A00-13033           Sample Status : A
Login by        : SDM005              Date Logged in: [redacted]
4:17:18
Auth/X by       : BATCH               Date Auth/X   : [redacted]
3:03:52
Submitter       : SDM005              Project Number: PPROP-004

Q Prod. Name    :                     Lot Number    :

Customer Name   :                     Sample Type   : HDPE

Sample Form     : PELLETS             Notebook #    :

Reason Code     : PROD_DEV            Sample Name   : HDPE-12

Job Name        : SDM_NANO_HDPE_7-12  Hazard        : 0-1-0
Description of Problem : ( SEE BELOW )
Please run GPC and DORS (Y-value) send results in excel spreadsheet
```

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	33280 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9693 Grams/cc
	A	T	Notebook	wxw-2-40
ADORS /1	A	T	RDR	1.59
	A	N	a value	3.2
		N	ab value	0.72

```
        |A |
|                |N | % error                  | 0.67
        |A |
|                |N | Test Temp                | 190 deg_C
        |A |
|                |N | % Strain                 | 20
        |A |
|                |N | Plate Size               | 25 mm
        |A |
|                |N | ETA*100                  | 10300.0 poise
        |A |
|                |T | ER                       | 1.99
        |A |
|                |T | PDR                      | 9.17
        |A |
|                |T | ET                       | NR
        |A |
|                |T | YR                       | 1006
        |A |
|                |N | Slope (ETA-0,GAMMA DOT-0 | 1.00
        |A |
|                |T | Run #                    | HDPE1000
        |A |
|                |T | Comment                  | PAH100300
        |A |
|AFR1902160/1    |N | Flow rate                | 1.72 Grams/10 m
        |A |
|                |T | Notebook                 | BRH4-139
        |A |
|AFR190HL   /1   |N | Flow rate                | 89.0 Grams/10 m
        |A |
|                |T | Notebook                 | BRH4-138
        |A |
|AGPC       /1   |T | Comment                  | results sent to submitte
r       |A |
-----------------------------------------------------------------------
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```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

[redacted] at 07:53 by SDMEHTA

Sample : A00-13024
Login by : SDM005
3:54:19
Auth/X by : BATCH
3:03:49
Submitter : SDM005

Sample Status : A
Date Logged in: [redacted]
Date Auth/X : [redacted]
Project Number: PPROP-004

Q Prod. Name :

Lot Number :

Customer Name :

Sample Type : HDPE

Sample Form : PELLETS

Notebook # :

Reason Code : PROD_DEV

Sample Name : HDPE-C1-ZSK30

Job Name : SDM_NANO_HDPE_7-12

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	R	Component Name	Result	St
AASH /1	N	Ash content	40 PPM	A
	T	Notebook	WXW-2-35	A
ADENSANEX /1	N	Average density	0.9590 Grams/cc	A
	T	Notebook	WXW-2-39	A
ADORS /1	T	RDR	1.84	A
	N	a value	3.0	A
	N	ab value	0.72	

```
      |A |
|            |N | % error                    | 0.54
      |A |
|            |N | Test Temp                  | 190 deg_C
      |A |
|            |N | % Strain                   | 20
      |A |
|            |N | Plate Size                 | 25 mm
      |A |
|            |N | ETA*100                    | 9910.0 poise
      |A |
|            |T | ER                         | 2.12
      |A |
|            |T | PDR                        | 8.97
      |A |
|            |T | ET                         | NR
      |A |
|            |T | YR                         | 795.2
      |A |
|            |N | Slope (ETA-0,GAMMA DOT-0 | 1.00
      |A |
|            |T | Run #                      | HDPE1000
      |A |
|            |T | Comment                    | PAH100300
      |A |
|AFR1902160/1 |N | Flow rate                 | 1.70 Grams/10 m
      |A |
|            |T | Notebook                   | BRH4-124
      |A |
|AFR190HL   /1 |N | Flow rate                | 74.6 Grams/10 m
      |A |
|            |T | Notebook                   | BRH4-123
      |A |
|AGPC       /1 |T | Comment                  | results sent to submitte
r     |A |
-----------------------------------------------------------------------
----------
```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

RESULT LIST

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-13025

[redacted] at 07:54 by SDMEHTA

Sample : A00-13025
Login by : SDM005
3:55:36
Auth/X by : BATCH
3:03:50
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV

Job Name : SDM_NANO_HDPE_7-12

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-004

Lot Number :

Sample Type : HDPE

Notebook # :

Sample Name : HDPE-C2-ZSK30

Hazard : 0-1-0

Description of Problem : (SEE BELOW)
Please run GPC and DORS (Y-value) send results in excel spreadsheet

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	100 PPM
	A	T	Notebook	WXW-2-35
ADENSANEX /1	A	N	Average density	0.9578 Grams/cc
	A	T	Notebook	WXW-2-39
ADORS /1	A	T	RDR	.95
	A	N	a value	2.6
		N	ab value	0.72

```
      |A |
|              |N | % error                    | 1.56
      |A |
|              |N | Test Temp                  | 190 deg_C
      |A |
|              |N | % Strain                   | 20
      |A |
|              |N | Plate Size                 | 25 mm
      |A |
|              |N | ETA*100                    | 22100.0 poise
      |A |
|              |T | ER                         | 1.62
      |A |
|              |T | PDR                        | 4.38
      |A |
|              |T | ET                         | NR
      |A |
|              |T | YR                         | 600.0
      |A |
|              |N | Slope (ETA-0,GAMMA DOT-0   | 1.00
      |A |
|              |T | Run #                      | HDPE1000
      |A |
|              |T | Comment                    | PAH100300
      |A |
|AFR1902160/1  |N | Flow rate                  | 0.578 Grams/10 m
      |A |
|              |T | Notebook                   | BRH4-124
      |A |
|AFR190HL   /1 |N | Flow rate                  | 16.3 Grams/10 m
      |A |
|              |T | Notebook                   | BRH4-123
      |A |
|AGPC       /1 |T | Comment                    | results sent to submitte
r     |A |
|AGPC       /2 |T | Comment                    | results sent to submitte
r     |A |
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Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.
```

RESULT LIST

Exhibit 9

EQUISTAR

INVENTION DISCLOSURE

CONFIDENTIAL

To be Completed by Patent Department

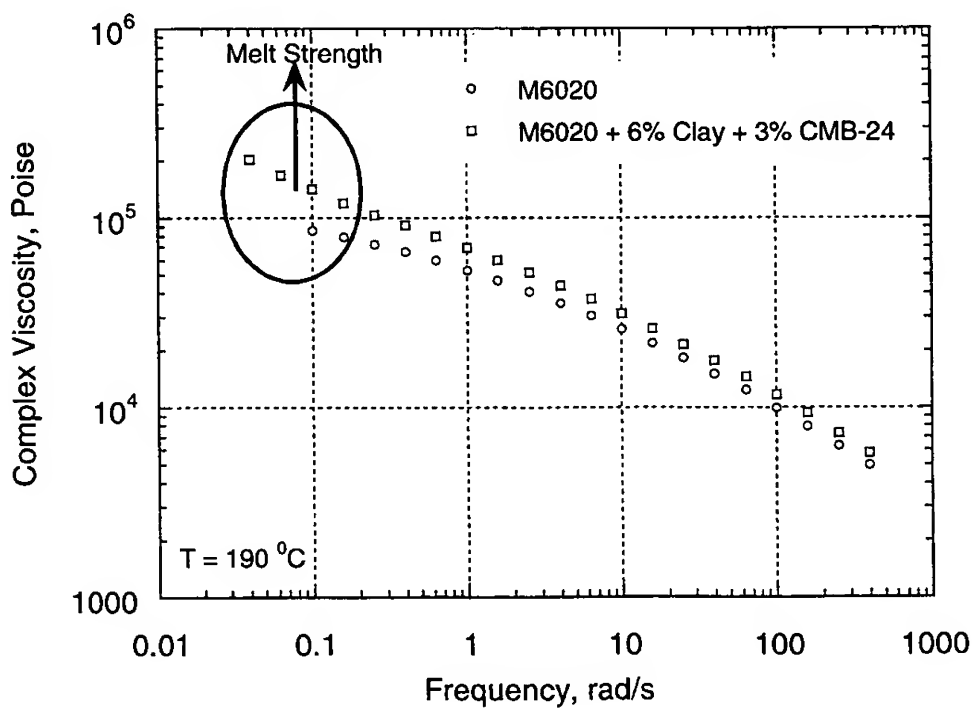
DISCLOSURE NO.: [redacted]
Assigned to: HDPE/W. Heidrich
Date Received: [redacted]

TITLE: High Density Polyethylene Nanocomposites with High Melt Strength

DESCRIPTION: (Please attach any sketches, pertinent reports, notebook pages, etc.)
HDPE based nanocomposites with 6% Treated Clay (Cloisite 15A) and 3% CMB-24 (maleic anhydride grafted HDPE) show an upturn in the complex viscosity at low frequencies (as indicated by dynamic oscillatory rheology measurements). This may be an indication of improved melt strength and may give us the potential to develop high melt strength PE resins. Some potential applications for this material include sheet, thermorforming, blow molding, foaming and extrusion coating. This might also give us the potential to develop high melt strength resins out of Matagorda (MTO). MTO resins are made using a Ziegler Natta catalyst and do not have as high melt strength as compared to chrome catalyzed HDPE resins (which have long chain branching).

We suspect that this upturn in viscosity at low shear rates is coming from the formation of a network structure due to an interaction between the clay platelets, the maleic anhydride grafted PE and the resin matrix. This network structure breaks down at processing shear rates and hence the nanocomposite based resin processes very similar to the base resin. The Figure below shows the upturn in our homopolymer HDPE (M6020).

Rheology Data for HDPE based Nanocomposites



EQUISTAR

CONFIDENTIAL

10/11/00

Important: You may lose the right to a patent if you publish, commercially us , offer to sell, disclose or show your invention to outsiders before a patent application is filed. Describ any such activities and give dates.			
Signature	Printed Name	Location/Phone	Date
SMehta	SAMEER MEHTA	ETC / 513-530-4161	[redacted]

Read and understood by: Edward S. Vaughn Date: [redacted]

Please e-mail the completed form and forward a signed and witnessed paper copy with any attachments to: Margaret Bailey, ETC-MSN53.

Exhibit 10

[redacted] 09:44 by SDMEHTA

Sample : A00-15263
Login by : SDM005
8:03:43
Auth/X by : BATCH
3:02:08
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV
MI, 0.91
Job Name : SDM_NANO_HDPE
Description of Problem : (SEE BELOW)
Please run DORS send data in Excel

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-003

Lot Number :

Sample Type : LLDPE

Notebook # :

Sample Name : HDPE-13 (0.7

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	38926 PPM
	A	T	Comments	N/A
	A	T	Notebook	CDR2/331
ACMLLDPE /1	A	T	Notebook	BRH5-214
ADENSANEX /1	A	N	Average density	0.9455 Grams/cc
	A	T	Notebook	wxw-2-105
ADORS /1	A	T	RDR	NR - Error too high.
	A	N	a value	2.8

```
|                    |N | ab value                    | 0.72
       |A |
|                    |N | % error                     | 5.01
       |A |
|                    |N | Test Temp                   | 150 deg_C
       |A |
|                    |N | % Strain                    | 20
       |A |
|                    |N | Plate Size                  | 25 mm
       |A |
|                    |N | ETA*100                     | 27800.0 poise
       |A |
|                    |T | ER                          | NR - Cor. coeff. too low
       |A |
|                    |T | PDR                         | 6.43
       |A |
|                    |T | ET                          | NR
       |A |
|                    |T | YR                          | NR
       |A |
|                    |N | Slope (ETA-0,GAMMA DOT-0    | 1.00
       |A |
|                    |T | Run #                       | LLDPE1000 ARES II
       |A |
|                    |T | Comment                     | PAH111500
       |A |
|AFLEXMOD   /1       |N | Crosshead speed             | 0.50 In/min
       |A |
|                    |N | 1% Secant modulus           | 69000 PSI
       |A |
|                    |N | 1% within sample std dev    | 737
       |A |
|                    |N | 2% Secant modulus           | 56100 PSI
       |A |
|                    |N | 2% within sample std dev    | 622
       |A |
|                    |T | Notebook                    | BRH5-238
       |A |
|AFR1902160/1        |N | Flow rate                   | 0.583 Grams/10 m
       |A |
|                    |T | Notebook                    | coop1-137
       |A |
|AFR190HL   /1       |N | Flow rate                   | 21.2 Grams/10 m
       |A |
|                    |T | Notebook                    | coop1-139
       |A |
----------------------------------------------------------------------
----------
```

cont'd....

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-15263
Page 2

```
------------------------------------------------------------------------
----------
| Analysis    |R |     Component Name      |               Result
      |St|
------------------------------------------------------------------------
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```

Notes:
All entered results have their current status displayed
status A is authorised
R is rejected
M is result entered and modified
E is result entered
X is cancelled.

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-15262
Page 1

[redacted] 09:43 by SDMEHTA

Sample : A00-15262
Login by : SDM005
8:00:26
Auth/X by : BATCH
3:02:07
Submitter : SDM005

Q Prod. Name :

Customer Name :

Sample Form : PELLETS

Reason Code : PROD_DEV
ol (ZSK3
Job Name : SDM_NANO_HDPE
Description of Problem : (SEE BELOW)
Please run DORS send data in Excel

Sample Status : A
Date Logged in: [redacted]

Date Auth/X : [redacted]

Project Number: PPROP-003

Lot Number :

Sample Type : LLDPE

Notebook # :

Sample Name : GATR311 Contr

Hazard : 0-1-0

These results are subject to verification and approval.

Analysis	St	R	Component Name	Result
AASH /1	A	N	Ash content	7547 PPM
	A	T	Comments	N/A
	A	T	Notebook	CDR2/331
ACMLLDPE /1	A	T	Notebook	BRH5-214
ADENSANEX /1	A	N	Average density	0.9233 Grams/cc
	A	T	Notebook	wxw-2-105
ADORS /1	A	T	RDR	1.40
	A	N	a value	2.3

```
|                |N | ab value                      | 0.72
       |A |
|                |N | % error                       | 2.03
       |A |
|                |N | Test Temp                     | 150 deg_C
       |A |
|                |N | % Strain                      | 20
       |A |
|                |N | Plate Size                    | 25 mm
       |A |
|                |N | ETA*100                       | 24900.0 poise
       |A |
|                |T | ER                            | 1.44
       |A |
|                |T | PDR                           | 4.23
       |A |
|                |T | ET                            | NR
       |A |
|                |T | YR                            | NR
       |A |
|                |N | Slope (ETA-0,GAMMA DOT-0      | 1.00
       |A |
|                |T | Run #                         | LLDPE1000
       |A |
|                |T | Comment                       | PAH111500
       |A |
|AFLEXMOD    /1  |N | Crosshead speed               | 0.50 In/min
       |A |
|                |N | 1% Secant modulus             | 57400 PSI
       |A |
|                |N | 1% within sample std dev      | 1587
       |A |
|                |N | 2% Secant modulus             | 48100 PSI
       |A |
|                |N | 2% within sample std dev      | 1206
       |A |
|                |T | Notebook                      | BRH5-237
       |A |
|AFR1902160/1    |N | Flow rate                     | 0.827 Grams/10 m
       |A |
|                |T | Notebook                      | coop1-137
       |A |
|AFR190HL    /1  |N | Flow rate                     | 22.5 Grams/10 m
       |A |
|                |T | Notebook                      | coop1-139
       |A |
----------------------------------------------------------------------
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```

cont'd....

EQUISTAR CONFIDENTIAL - Result list for sample no. A00-15262
Page 2

```
--------------------------------------------------------------------------
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| Analysis     |R |       Component Name        |              Result
      |St|
--------------------------------------------------------------------------
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```

Notes:
  All entered results have their current status displayed
    status A is authorised
           R is rejected
           M is result entered and modified
           E is result entered
           X is cancelled.

Exhibit 11

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INVENTION DISCLOSURE

GB
WH
MB
DS
FILE

RECEIVED

[redacted]

PATENT

To be Completed by Patent Department
DISCLOSURE NO.: [redacted]
Assigned to: LLDPE/W. Heidrich
Date Received: [redacted]

TITLE: Linear Low Density Polyethylene Nanocomposites with High Melt Strength

DESCRIPTION: (Please attach any sketches, pertinent reports, notebook pages, etc.)
LLDPE based nanocomposites with 6% treated clay and 3% maleic anhydride grafted HDPE show an upturn in the complex viscosity at low frequencies (as indicated by dynamic oscillatory rheology measurements). This is an indication of improved melt strength and was confirmed by the sag test. This upturn may give us the potential to develop high melt strength LLDPE resins with good impact and tear properties.

We suspect that this upturn in viscosity at low frequencies (shear rates) is coming from the formation of a network structure due to an interaction between the clay platelets, the maleic anhydride grafted PE and the resin matrix. This network structure breaks down at processing shear rates and hence the nanocomposite based resin processes very similar to the base resin. Figure 1 shows the upturn in viscosity in our Petrothene Select LLDPE (GATR311, 0.7 MI, 0.917 density). Figure 2 shows the improvement in sag time with the GATR311 based nanocomposite.

FIGURE 1

Rheology Data on LLDPE (GATR311) Nanocomposites

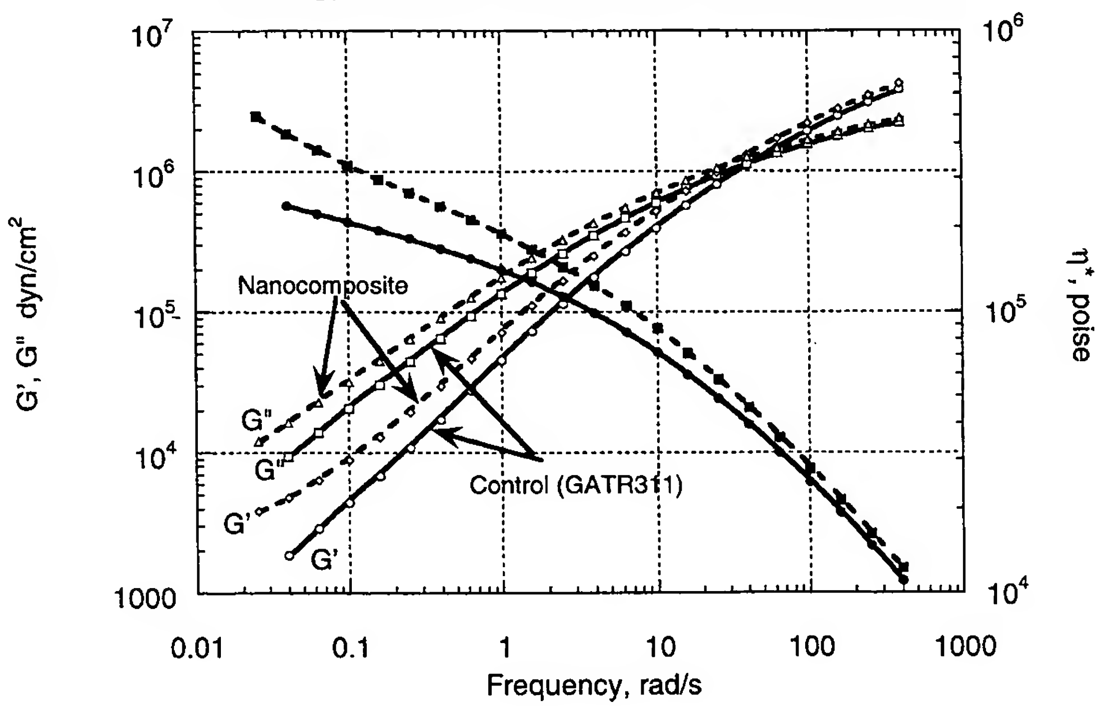

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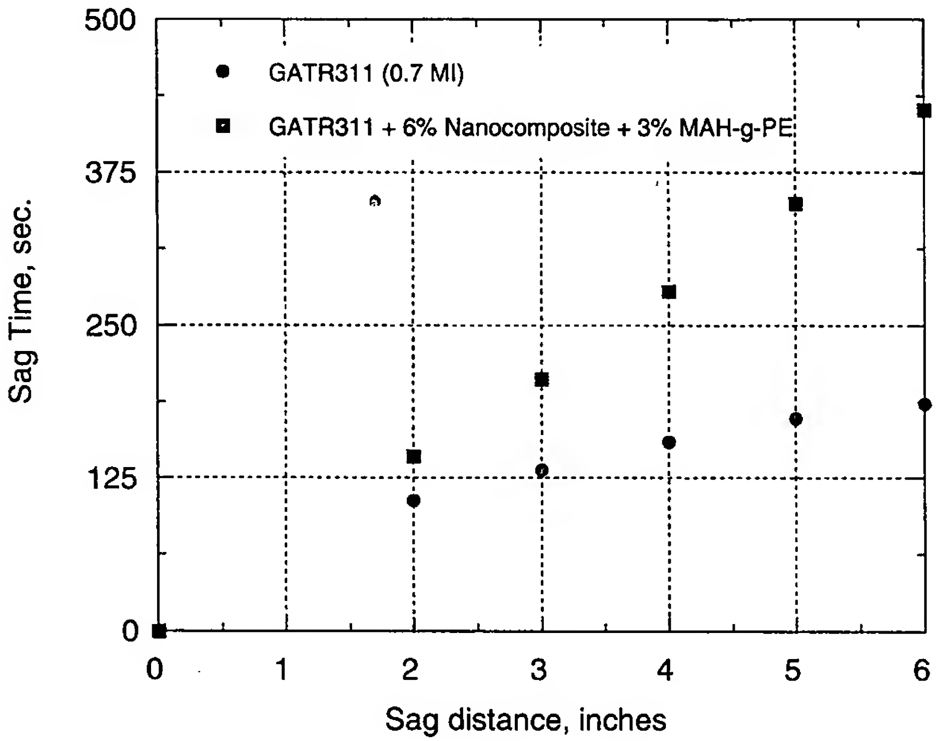
FIGURE 2

Sag Results for LLDPE at 190 C



Important: **You may lose the right to a patent if you publish, commercially use, offer to sell, disclose or show your invention to outsiders before a patent application is filed. Describe any such activities and give dates.**

Signature	Printed Name	Location/Phone	Date
S Mehta	SAMEER MEHTA	ETC /513-530-4161	[redacted]
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Edward S. Vargas	Edward S. Vargas	ETC/513-530-4121	[redacted]

Read and understood by: T Shanker Narayanan **Date:** [redacted]

Please e-mail the completed form and forward a signed and witnessed paper copy with any attachments to:
Margaret Bailey, ETC-MSN53.